

Page
①

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 09-308822

(43)Date of publication of application : 02.12.1997

(51)Int.Cl.

B01J 13/00
A61K 7/00
A61K 9/107
A61K 47/34
B01F 17/38
B01F 17/56
// A23D 7/00

(21)Application number : 08-126661

(71)Applicant : HENKEL JAPAN KK

(22)Date of filing : 22.05.1996

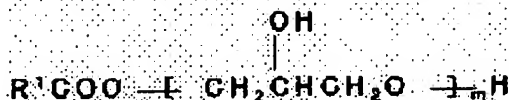
(72)Inventor : MICHİYOSHI KADOFUMI
TOTANI EISEI

(54) OIL-IN-WATER TYPE EMULSION AND ITS PRODUCTION

(57)Abstract:

PROBLEM TO BE SOLVED: To prepare a stable and fine emulsion which does not generate formalin and does not cause pH alteration due to deposition after long time storage by blending polyglycerin ester having a specified formula, alkyl polyglycoxide having a specified formula, and an oil agent.

SOLUTION: At the time of producing a fine oil-in-water type emulsion to be used for medical products, cosmetic products, etc., one or more kinds of polyglycerin ester of the formula (in the formula, R₁ is a 7-21C straight chain or branched chain, saturated or unsaturated aliphatic group; (m) is an integer of 4-15), an alkyl polyglycoxide having the formula R₂OGr_n (in the formula, R₂ is a 8-22C straight chain or branched chain alkyl or alkenyl group; (n) is an integer of 1-4), and one or more kinds of oil agents. In this case, 2-8wt.% of a polyglycerin ester, 0-5wt.% of an alkyl polyglycoxide, and 1-40wt.% of an oil agent are mixed.



LEGAL STATUS

Page 2

[Date of request for examination] 22.05.2003

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

Page 3

* NOTICES *

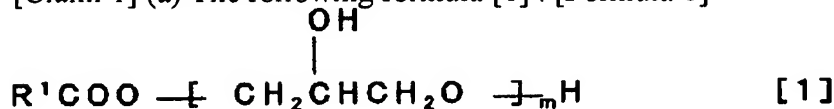
JPO and INPIT are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] (a) The following formula [1] : [Formula 1]



One sort of the polyglycerin ester expressed with (as for the inside R1 of a formula the fat radical of the saturation of the straight chain of a carbon number 7-21 or branched chain or partial saturation is shown, and m shows the number used as 2-15) or two sorts or more, and the formula of (b) following [2]: It is [Formula 2].

R²OG_n

[2]

It is the oil-in-water type emulsification object [claim 2] characterized by blending one sort of the alkyl poly glycoside expressed with (as for the inside R2 of a formula the alkyl group or alkenyl radical of the straight chain of a carbon number 8-22 or branched chain is shown, and n shows the number used as 1-4), and (c) oiliness agent, or two sorts or more. The emulsification object of claim 1 with which 2 to 8 % of the weight and alkyl poly glycoside (b) are characterized by 0 to 5 % of the weight and an oily agent (c) existing in 1 to 40% of the weight of an amount by polyglycerin ester (a).

[Claim 3] The oil phase which made the oily agent (c) dissolve or distribute polyglycerin ester (a) according to claim 1 and alkyl poly glycoside (b) is heated beyond phase inversion temperature (80 degree-85°C), and is agitated. ** et al., The manufacture approach of the oil-in-water type emulsification object characterized by adding gradually the aqueous phase heated to the same temperature (80 degree-85°C), making it emulsify, and cooling and carrying out phase inversion to below phase inversion temperature (below 40-degreeC).

[Translation done.]

* NOTICES *

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the stable oil-in-water type emulsification object generally widely used for the industrial application of food, drugs, cosmetics, and others etc., and its manufacture approach. Furthermore, it is related with the approach of manufacturing a stable and detailed emulsification object, by the usual churning without after a mothball almost not having generating of formalin or the change of pH by decomposition and needing special equipments, such as the conventional **** high-speed agitator, on the occasion of manufacture in detail.

[0002]

[Description of the Prior Art] When preparing conventionally the detailed oil-in-water type emulsification object used for food, drugs, cosmetics, etc., in order to choose the surfactant suitable for the oily agent emulsified and to distribute an oil phase and the aqueous phase as finely as possible, special machine equipments, such as a disperser (a homogenizer and colloid mill) of high shear and a high voltage emulsifier, are used.

[0003] Moreover, the preparation approach of the stable oil-in-water type emulsification object of an emulsification condition is considered by the phase inversion emulsification method recently. In order to prepare an oil-in-water type emulsification object with a phase inversion emulsification method, once choosing the surfactant which suited the emulsifying-oil-ed nature agent, dissolving this in an oily agent and considering as a water-in-oil type emulsification condition, the aqueous phase is added, phase inversion is carried out to an oil-in-water type emulsification object, and a stable emulsification object is obtained.

[0004] To JP,7-173380,A, phase inversion emulsification is tried using polyglycerin ester. In this case, although polyglycerin ester with HLB high as a surfactant which suited is desirable, when the polyglycerin of isolation generally remains in the high polyglycerin ester of HLB, therefore polyglycerin ester is distributed in an oily agent, the polyglycerin ester of high HLB which gel insoluble matter generated, and removed isolation polyglycerin beforehand since it was unsuitable to phase inversion emulsification is prepared, and the stable oil-in-water type emulsification object is prepared with the phase inversion emulsification method using this. However, in case phase inversion emulsification is carried out, in spite of having emulsified using high-speed ***** of 3000r.p.m, 5.6% and 3 hours after, the rate of water-repelling shows 12%, and an emulsification object cannot say it with it being not much stable 1 hour after ** et al. and here.

[0005] About the phase inversion temperature emulsification method, fiesta (T. Foerster) etc. gave 16 International journal OBU cosmetic Science and explanation detailed to 84 pages (1994), this predicted the phase inversion temperature of the formula and system of the suitable emulsifier for an emulsifying-oil-ed nature agent, it was checked by experiment, and it is said that the count approach is right in approximation. However, in order to control HLB of an emulsifier delicately in the reference of *****, the nonionic active agent of polyethylene glycol ether or an ester mold is used.

[0006] Thus, using polyethylene glycol ether or an ester mold nonionic active agent has the fault that

disassemble the emulsification object during a mothball, and generate formalin or pH falls. Moreover, the paraben currently widely used for cosmetics etc. as antiseptics has the problem that stick to the part of the ethylene glycol chain of this activator, and that effectiveness falls.

[0007] Although the approach of emulsifying using poly glycerol branching fatty acid ester is described in order to avoid generating of formalin and the lowering of pH by the decomposition under preservation like the above to JP,58-185537,A and to maintain the effectiveness of paraben antiseptics further, homogeneity is made to carry out emulsification distribution using a homomixer also in this case. Although generating of formalin and the change of pH under preservation of an emulsification object are considered here, the stability of an emulsification condition is only indicated to be only stability for three months by 5-degreeC-40°C, and neither the particle diameter of an emulsion nor the rate of water-repelling is displayed.

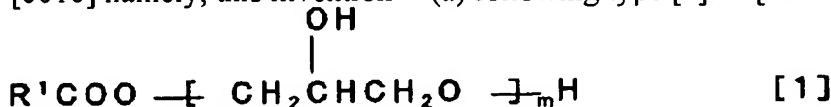
[0008]

[Problem(s) to be Solved by the Invention] The oil-in-water type emulsification object indicated by above-mentioned reference and an above-mentioned official report deteriorates by the mothball, and when generating formalin or preparing what has a problem in the chemical stability of pH falling, and an emulsification object, it must use high-speed churning equipment. Therefore, stability was physically [chemically and] good during the mothball, and a detailed oil-in-water type emulsification object which can be prepared with an easy agitator was desired.

[0009]

[Means for Solving the Problem] In order to solve this problem, as a result of inquiring wholeheartedly, after this invention person etc. blended alkyl poly glycoside with polyglycerin ester by the specific ratio and dissolved or distributed the desired oily agent, he invented attaining the above-mentioned object by adding the aqueous phase with a phase inversion temperature emulsification method, and stirring.

[0010] namely, this invention -- (a) following type [1]: -- [Formula 3]



The formula of one sort or two sorts or more, and (b) following of the polyglycerin ester expressed with (as for the inside R1 of a formula the fat radical of the saturation of the straight chain of a carbon number 7-21 or branched chain or partial saturation is shown, and m shows the number used as 4-15)

[2]: It is [Formula 4].



The oil-in-water type emulsification object characterized by blending one sort of the alkyl poly glycoside expressed with (as for the inside R2 of a formula the alkyl group or alkenyl radical of the straight chain of a carbon number 8-22 or branched chain is shown, and n shows the number used as 1-4) and (c) oiliness agent or two sorts or more and its manufacture approach are offered.

[0011] Furthermore, polyglycerin ester (a) and alkyl poly glycoside (b) are made to dissolve or distribute in an oily agent (c) in detail. The approach of adding gradually the aqueous phase which heated beyond the phase inversion temperature set up with the phase inversion temperature emulsification method (80 degree-85°C), agitated, and was heated to the same temperature as ** et al. and an oil phase, agitating it, cooling below to ** phase inversion temperature (below 40-degreeC), and manufacturing an oil-in-water type emulsification object is offered.

[0012] As for polymerization degree, 2-15, especially 4-10 are desirable with the ester of the polyglycerin which carried out the polymerization of the polyglycerin ester used by this invention, and a fatty acid. The isooctane acid which has a caprylic acid, a capric acid, a lauric acid, a myristic acid, a palmitic acid, palmitoleic acid, stearic acid, oleic acid, linolic acid, behenic acid, and branched chain, an iso decanoic acid, isostearic acid, etc. are used, and the class of fatty acid esterified is the monoester. Although these polyglycerin ester can obtain various ester from a hydrophilic property to oleophilic (HLB:2-16) by changing whenever [polymerization-degree / of a glycerol /, and chain length / of a fatty

acid / or esterification], to manufacture of an oil-in-water type emulsification object, its comparatively high thing of HLB is desirable. Sakamoto Pharmaceutical industry is marketing such polyglycerin ester by the trade name of SY Glyster.

[0013] Since polyglycerin ester does not contain an ethylene oxide chain in a molecule, even if it saves the emulsification object which used this for a long period of time, it is dramatically rare to generate formalin by decomposition or to cause change of pH. Moreover, there are few phenomena in which antiseptics, such as paraben, adsorb and decrease in number the effectiveness.

[0014] The alkyl poly glycoside used for this invention is indicated by many official reports at a U.S. Pat. No. 3547828 number, said 3839318 numbers, the Europe patent application EP No. 301298, said 362671 numbers, and the German patent official report No. 3927919. Alkyl poly glycoside is manufactured by either of the transformer acetalization using the thing which carried out the direct reaction of superfluous alcohol and a superfluous catalyst to the glucose or a solvent, and lower alcohol.

[0015] The alkyl poly glycoside used for this invention is the resultant of the alcohol which has reducing sugars, such as a glucose, a fructose, a mannose, a galactose, arabinose, and a xylose, and eight to 22 carbon atoms, and reducing sugar of each other may be combined by the glycosidic linkage. The number of reducing sugars united [each other] is shown as n in a formula [2]. Generally polymerization degree n is between the value of 1-3, especially 1.1-1.4. The reducing-sugar unit guided from the reducing sugar mentioned above is expressed by G of a formula [2]. R2 in a formula [2] is guided from the alcohol of a straight chain or branched chain by the alkyl or the alkenyl radical which has the carbon number of eight to 22 pieces. The poly glycoside which had the alkyl group of 8-16 especially is desirable for the object of this invention. Such alkyl poly glycosides are marketed by Henkel Hakusui, Inc. by the trade name of guru KOPON or a planter care.

[0016] Although it changes with the applications as an oily agent used for this invention, liquid paraffin, paraffin wax, vaseline, squalene, squalane, lanolin, higher-alcohol, and higher-alcohol-fatty acid ester, fatty acid ester, a fatty-acid monoglyceride, diglyceride, a triglyceride, natural animal and plant oil, silicone oil, etc. are used.

[0017] The formula which suited the oily agent of the request used for the report (International journal OBU cosmetic Science) of the fiesta of the above-mentioned [polyglycerin ester, the class of alkyl poly glycoside, and a compounding ratio] etc. according to the sequence of a publication is calculated. That is, the weighted average efficiency of the alkene carbon number (ACN) of the hydrocarbon equivalent to the oily agent mixture used first is calculated, then emulsifier mixture (polyglycerin ester and alkyl poly glycoside) is selected, desired phase inversion temperature is set up, and an emulsifier formula is calculated using the phase inversion temperature (HFxEACNo) and the slope factor (HF) of a proper of each emulsifier. The particle diameter and viscosity of an emulsification object are measured by experiment on the basis of the value, and the combination value of practical use is determined. the result of the above trial -- an oily agent -- it became clear to 1 to 40 % of the weight that polyglycerin ester has 0 to 5% of the weight of the desirable compounding ratio of 2 to 8 % of the weight and alkyl poly glycoside. When this range is surpassed, a problem is seen arise in stability, viscosity, etc. of an emulsification object.

[0018]

[Example] An example is given to below and this invention is explained to a detail.

[A table 1]

【表 1】

	本発明の配合例					比較例
成分	1	2	3	4	5	1
ポリオキシエチレン(12) セトステアリルエーテル モノステアリン酸 ^g リセリル アルキル ^g リコシト ^g (C12-16、重合度1.4)						3.9
ヘキサ ^g リセリンモノステアレート				4.7	5.0	2.1
ヘキサ ^g リセリンモノオレート			4.5			
ヘキサ ^g リセリンモノオレート				1.3		
2-エチルヘキサン酸セチル トリカ ^g リルカ ^g リン酸 ク ^g リセリト ^g ステアリン酸オクチル スクワラン	20.0	20.0	20.0	20.0	3.0 15.0 2.0	20.0
精製水	残余 (合計100とする)					
	調製直後					
粒子径(μm)	0.1	0.4	0.1	0.1	0.1	0.1
粘度(mPa·s)		全て100以下				100以下
pH	6.9	6.7	6.2	6.0	7.5	6.9
	40℃、30日後					
粒子径(μm)	0.1	0.4	0.1	0.1	0.1	0.1
安定性	分離なし	なし	なし	なし	なし	分離なし
pH	6.5	7.0	6.5	6.4	6.9	3.3

The emulsifier was added, the oily agent shown in a table 1 was agitated by the usual ***** (300r.p.m), and ** 80-degree-85°C was made to heat and distribute it for a beaker. Next, in addition, the water of the remainder after churning was added and emulsified for 5 minutes gradually [5 to 30 % of the weight of purified water which agitated this dispersed oil phase and preheated to ** 80 degree-85°C], and it cooled to 25-degreeC. After measuring immediately after manufacturing the particle diameter of the emulsification object prepared with the phase inversion temperature emulsification method using the formula of the example of combination of this invention, and the example of a comparison, viscosity, and pH and putting this emulsification object by 40-degreeC for 30 days, particle diameter, pH, and a water separation condition are measured, and that result is shown in the lower part of a table 1. Particle diameter was measured with the Shimadzu laser diffraction type particle-size-distribution measuring device (SALD-2000A).

[0019]

[Effect of the Invention] The oil-in-water type emulsification object of this invention is prepared with the easy usual agitator, and, also physically and chemically, is a good emulsification object of stability.

[Translation done.]